Interim Technical Report for First Year

on

Title of Project: "Development of Ceramic Laser Element"

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By Materials Team

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14. ABSTRACT
Practical laser generation from polycrystalline ceramic materials has become available; furthermore, it is expected that ceramic laser technology will completely surpass the conventional glass and single crystal laser technologies in the future. In this work development of high quality ceramic laser materials was successfully done by control on macro-, micro- and nano-structural refinement. Transparency and optical homogeneity were confirmed to be high enough for the development of high power laser generation. In addition optical defects such as residual pores and atomic defects were hardly observed in the fabricated ceramic laser elements.

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Abstract

Practical laser generation from polycrystalline ceramic materials has become available; furthermore, it is expected that ceramic laser technology will completely surpass the conventional glass and single crystal laser technologies in the future. In this work, development of high quality ceramic laser materials was successfully done by control on macro-, micro- and nano-structural refinement. Transparency and optical homogeneity were confirmed to be high enough for the development of high power laser generation. In addition, optical defects such as residual pores and atomic defects were hardly observed in the fabricated ceramic laser elements.

Keywords: Transparent ceramics, transparent polycrystalline material, ceramic laser, high energy laser, high power laser, engineered ceramic composite, waveguide

1. Introduction

Solid-state laser is widely used in metal processing, medical applications, such as eye surgery, red-green-blue (RGB) light sources in laser printer and projectors, environmental instrumentation measurements, optical transmission systems and future nuclear fusion process. Single crystal or glass as a laser gain medium is generally used in the solid-state laser, which was originated from ruby laser by Maiman in 1960. Since the success of Geusic *et al.* in generating continuous-wave (c.w.) laser oscillation using Nd:YAG single crystal at room temperature in 1964, solid-state laser using single crystal has been continuously developed up till now.

The first laser oscillation using transparent Nd:YAG ceramics was demonstrated by Ikesue *et al.* in the 1990s with laser performance comparable to single-crystal laser oscillation. Recently, ceramic laser technology has emerged as a promising candidate because of its numerous advantages over single-crystal lasers. First, ceramic can be produced in large size which is attractive for high-power laser generation. Second, it can be processed into fibre-laser gain medium for generating laser with high beam quality, and also be processed into composite laser gain medium with complicated structures which are principally difficult to fabricate by conventional single crystal technology. The development of ceramic laser technology has led to the achievements of compact and highly efficient laser oscillation, ease of control of laser mode, generation of coherence beam with high focusing property. Moreover, the realization of high-power laser based on ceramic technology has brought the visions of ultrahigh-speed machining technology and nuclear fusion one step closer.

2. Objective of the project

The objective of the project is to develop a ceramic gain media for laser generation of high output power and high beam quality. Materials team is to fabricate ceramic laser materials having high optical quality and special configurations which are difficult to achieve from the conventional single crystal by melt-growth method and glass materials. In addition, base points for the development of next generation solid laser will be constituted. Basic technology of ceramic waveguide element, which is controlled with micro-structural refinement and macro- structural design, will be developed, and with the aid of analysis team, laser performances for high power output lasers will be evaluated with related to the thermo mechanical properties of the interface of waveguide designed ceramic laser element.

3. Project schedule and targets

The project will proceed on the basis of the materials development technology by materials team. Materials development will be involved; (1) materials synthesis and designing for the recognition of new functionality and high power laser generation, (2) machining of those prepared materials and surface treatment, (3) evaluation of basic properties of those prepared materials. It is planned to run as a three-year research project as shown in Fig.1, and targets for each year term are summarized in table 1. In the first year, media with lowest scatter loss will be prepared, and special laser elements will be prepared in the second and third year to achieve high output power (~1kW) by focusing on waveguide designed media.

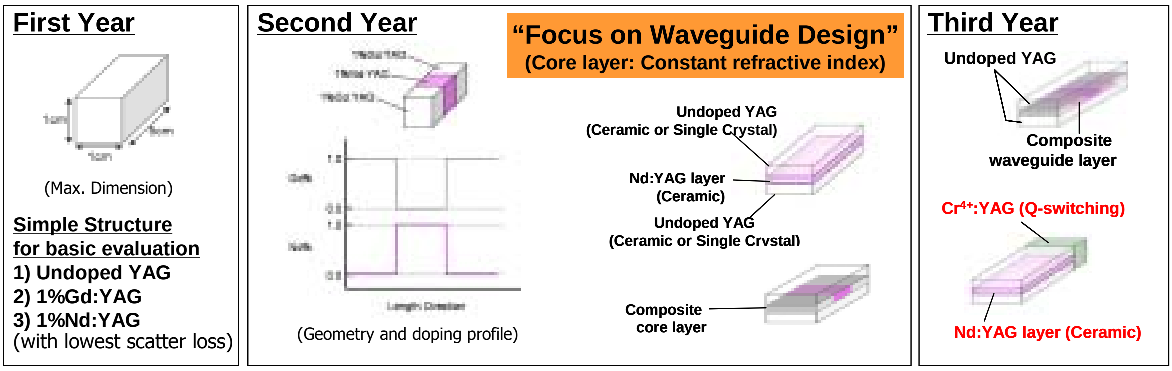


Fig 1. Laser gain media to be prepared in each year term of the project.

Table 1 Targets for each year term of the project

	Target Laser Output Power
First Year (2008)	Media with lowest scatter loss (~10^{-3}/cm)
Second Year (2009)	~100W
Third Year (2010)	300W ~ 1kW

4. Fabrication of high quality transparent ceramics

In the first year term, simple structure of undoped YAG, 1%Gd:YAG, and 1%Nd:YAG samples will be fabricated for basic evaluation. Laser gain media with lowest scattering loss will be prepared by modifying the ceramic process mainly on the microstructural refinement. The target of the optical quality of the materials is up to 10^{-3}/cm (0.001/cm) of scatter loss, which equal to the quality of commercialized Konoshima ceramic sample. Maximum dimension of each sample to be prepared for evaluation of basic properties is up to 1cmx1cmx5cm.

Solid-state reactive sintering process was applied to fabricate transparent ceramic laser gain media. Figure 2 shows the process, and figure 3 shows the SEM images of starting raw materials (high purity, 99.99%) used in this work. The primary particle sizes of Y2O3, Al2O3, and Nd2O3 were approximately 60nm, 300nm, and 400nm, respectively. These powders, blended with the stoichiometric ratio of garnet, were mixed and ball milled for one night in ethanol solvent with 0.5 mass% TEOS (tetraethyl orthosilicate) and a suitable amount of organic binder. The alcohol solvent was removed by spray drying the milled slurry, and at the same time granulated powders of about 50μm with spherical shapes were achieved.

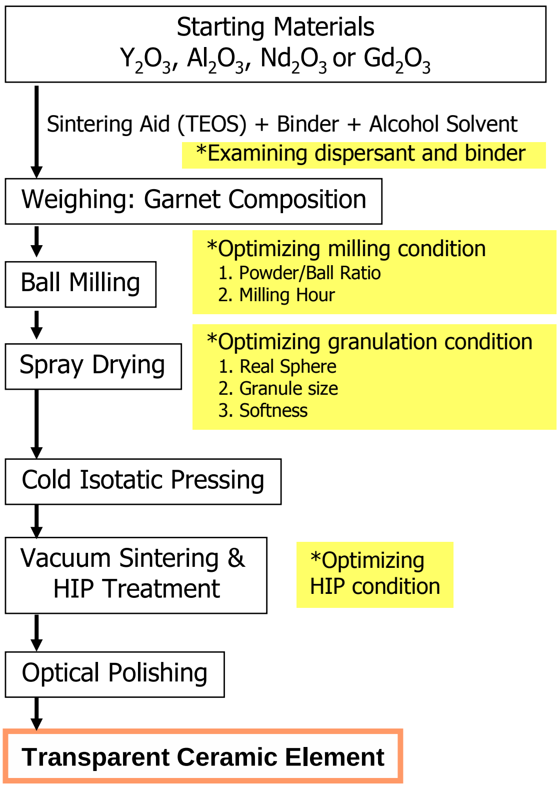


Fig.2 Fabrication process for the ceramic laser gain media.

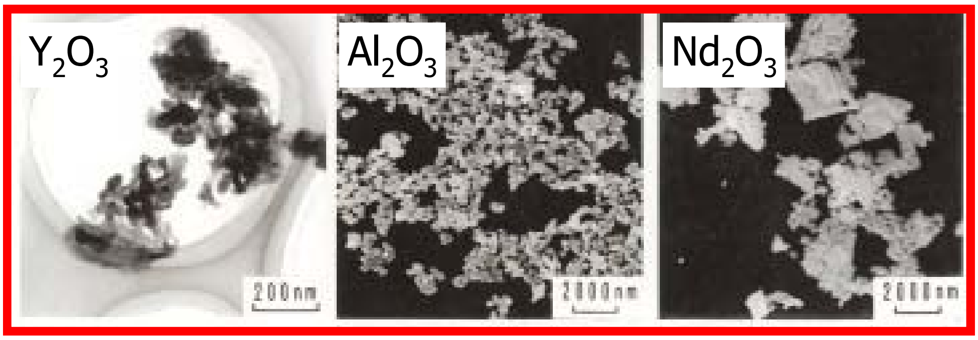


Fig.3 SEM images of the raw powder materials used as starting raw materials.

The spray-dried granulated powders were pressed with light pressure into a slab shape in a metal mold. The near-net-shaped sample was then cold isostatically pressed at 98–196 MPa, and a powder compact with a packing density of 50~55% of theoretical density was obtained. After heating of the pressed part to remove the organic component, the powder compact was sintered in vacuum (1×10^{-3} Pa) at 1750◦C for a few~twenty hours to obtain transparent Nd:YAG ceramics. In addition, HIP (hot isostatic pressing) treatment was performed to further eliminate residual pores. After polishing, transparent ceramic laser gain media were achieved.

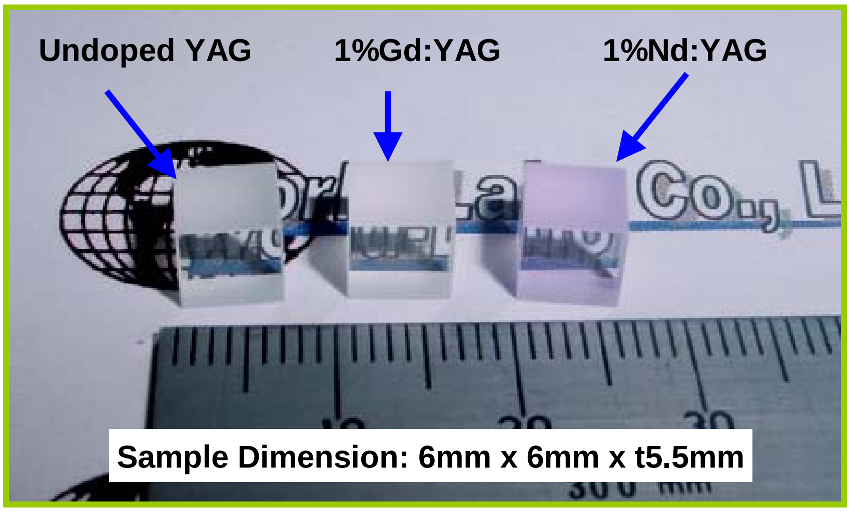


Fig.4 Appearance of fabricated samples for the evaluation of basic properties.

The transparent ceramic slabs (undoped YAG, 1%Gd:YAG, and 1%Nd:YAG) were machined into small blocks (dimension: 6mm x 6mm x t5.5mm) for optical measurements in macro level. The appearance of the fabricated samples is shown in Fig.4.

5. Observation in macro level

Polarized images of the whole samples are summarized in figure 5. Mechanical stress free condition was confirmed in all samples. Figure 6 shows the transmitted wavefront image of the all fabricated samples by interferometry. Almost straight fringes were observed, and this showed that the refractive index distribution in the whole sample is homogeneous.

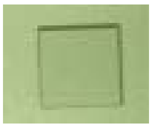
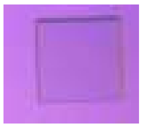
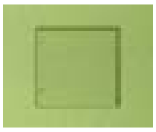
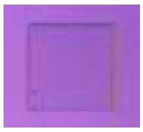
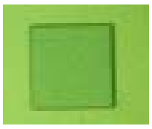
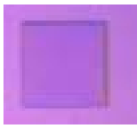
Materials	Open Nicol	Cross Nicol
Undoped YAG		
1%Gd:YAG		
1%Nd:YAG		

Fig.5 Polarized images of the fabricated transparent ceramic samples.

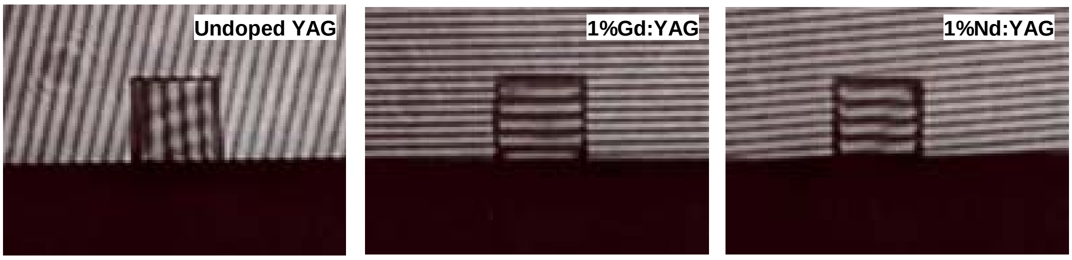


Fig.6 Transmitted wavefront image of the fabricated transparent ceramic samples.

In addition, in-line transmittance of each sample was measured from UV to visible and IR wavelength regions. Transmittance curves of each sample are shown in Fig.7. The in-line transmittance was as high as 84% transmittance, the theoretical value, which is equivalent to commercial 1.0 at.% Nd:YAG single crystal grown by the Czochralski method.

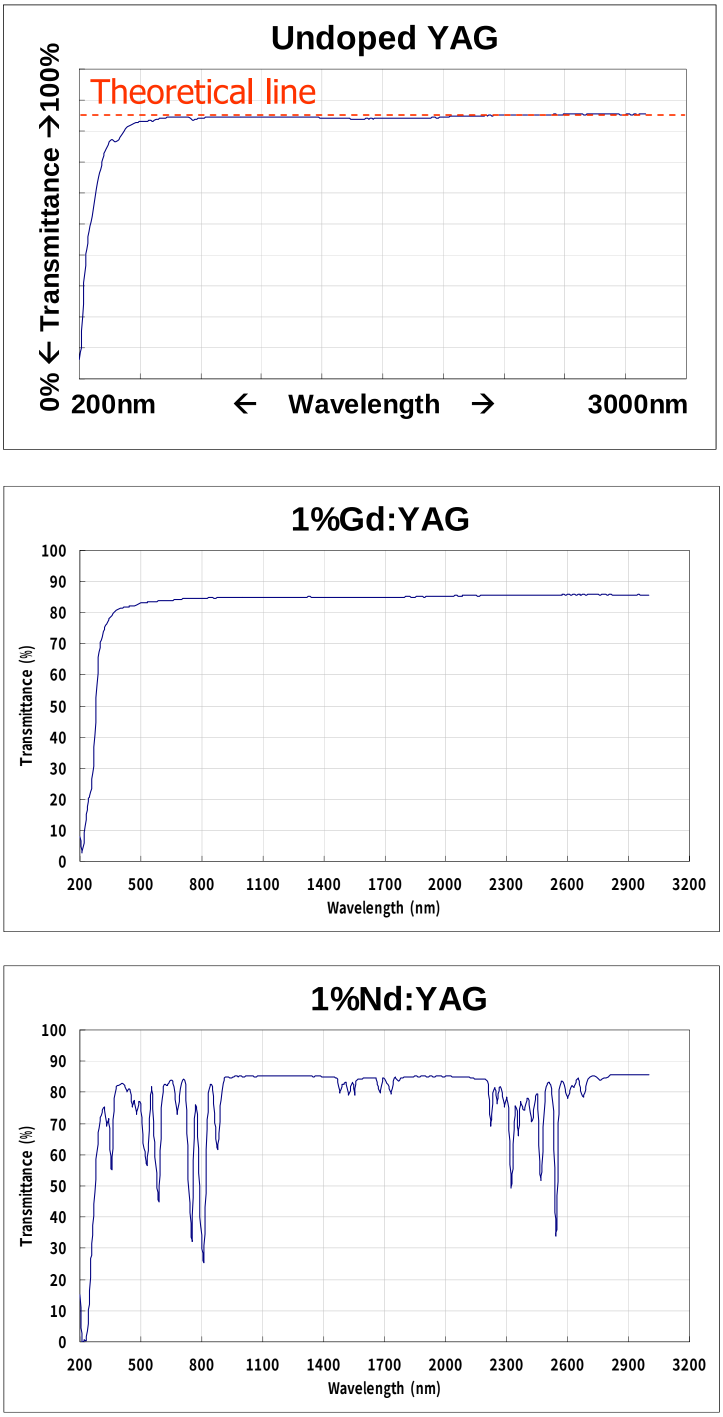


Fig.7 Transmittance curves of the fabricated transparent ceramic samples.

6. Observation in micro level

6.1 Under optical microscope (OM)

To check the grain size and microstructure of each transparent ceramics, one face polished samples were thermally etched at 1500C for 30 minutes, and they were observed under an optical microscope. Figure 8 shows the reflection microscopic images of thermal etched surface of each sample. Uniform microstructures and fine grain sizes of ~10μm level were confirmed.

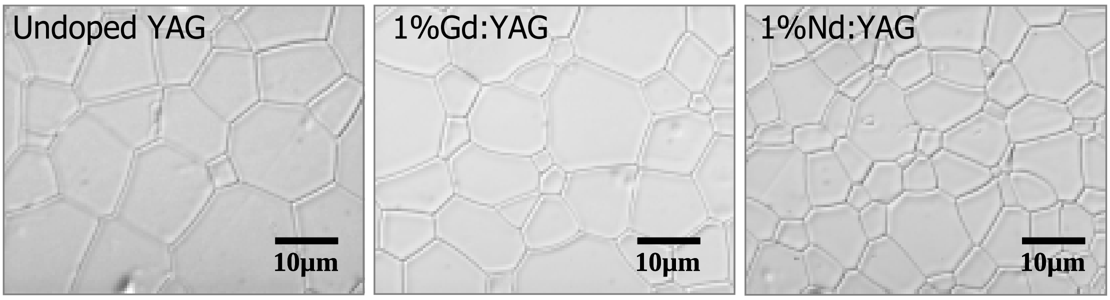


Fig.8 Thermal etched surfaces of the fabricated transparent ceramic samples observed under an optical microscope.

Figure 9 shows the transmitted polarized microscopy with open and cross nicol of each sample. No residual pores were observed, and very high optical homogeneity (birefringence free) was confirmed even at micron level.

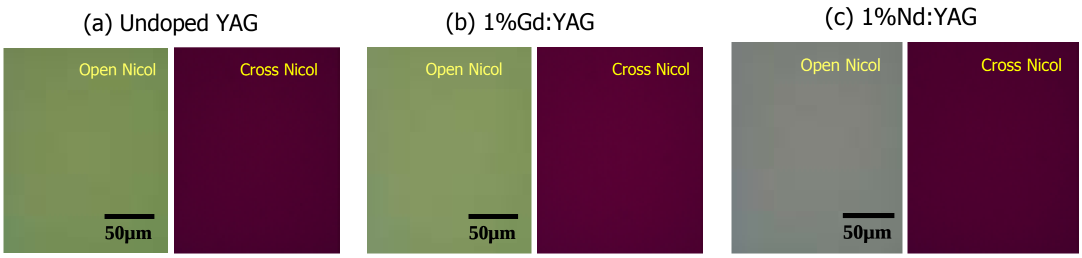


Fig.9 Transmitted polarized microscopy of the transparent ceramic samples.

6.2 By SEM and EPMA (electron probe micro analysis)

In order to check the internal grain size and microstructure, each of the sample were crushed down. Fracture surfaces of each sample were observed under SEM (see Fig.10). As observed under optical microscope, the internal microstructure was consistent with the surface microstructure. Moreover, almost no residual pores and no grain boundary phases were confirmed under SEM observation.

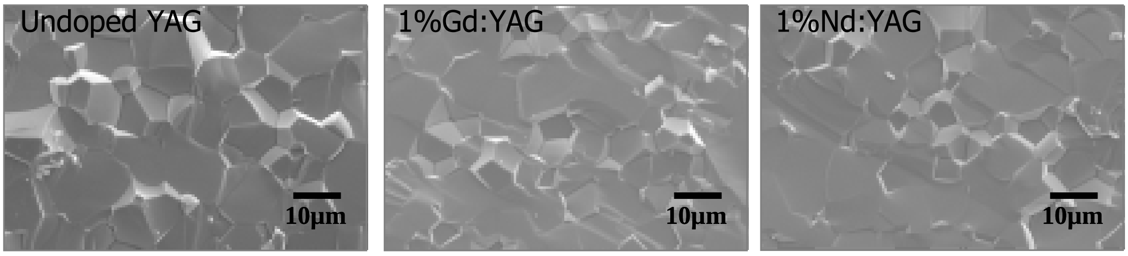


Fig.10 Fracture surfaces of the fabricated transparent ceramic samples by SEM observation.

Electron probe micro analysis was done for each sample in order to investigate the elemental (Y, Al, Gd, and Nd) distribution in micro level. The measured results were summarized in figure 11. It was confirmed that the dopant ions are homogeneously distributed and segregation of the dopant ions at macro-to-micro level was not observed.

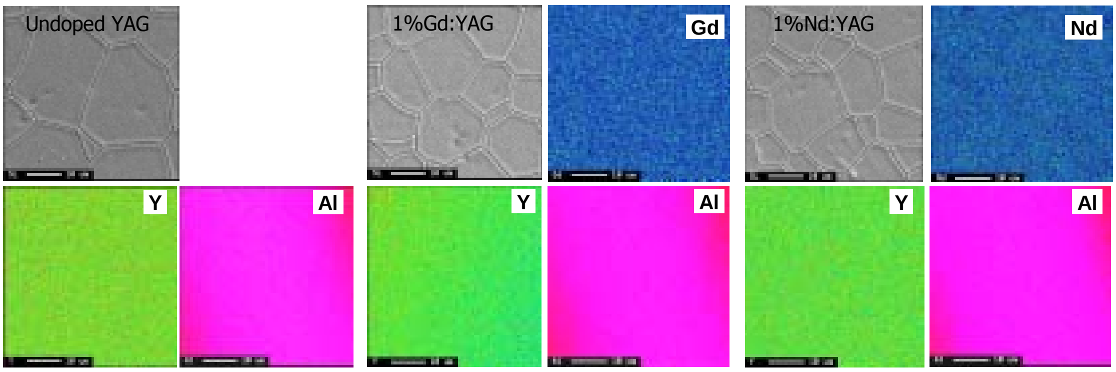


Fig.11 Electron probe micro analysis (EPMA) images of the fabricated transparent ceramic samples.

7. Observation in nano-level (High Resolution TEM analysis)

High resolution transmission electron microscopy was applied to investigate the lattice defects and formation of grain boundary phase was detected by using EDS (energy-dispersive spectroscopy) analysis. The results are summarized in figure 12. No atomic defects were recognized in the observed region of all samples. Clean grain boundaries (no secondary phases) were observed for all samples, except a few nano-meter thick amorphous layer (including SiO2 from the sintering aid TEOS) was detected in some regions of grain boundaries. However, the thickness (2~3nm) of the amorphous layer is sufficiently small compared to the wavelength of the target laser wavelength (1064nm from Nd:YAG). Therefore, it can be considered that it may hardly effect on the optical quality of transparent ceramics.

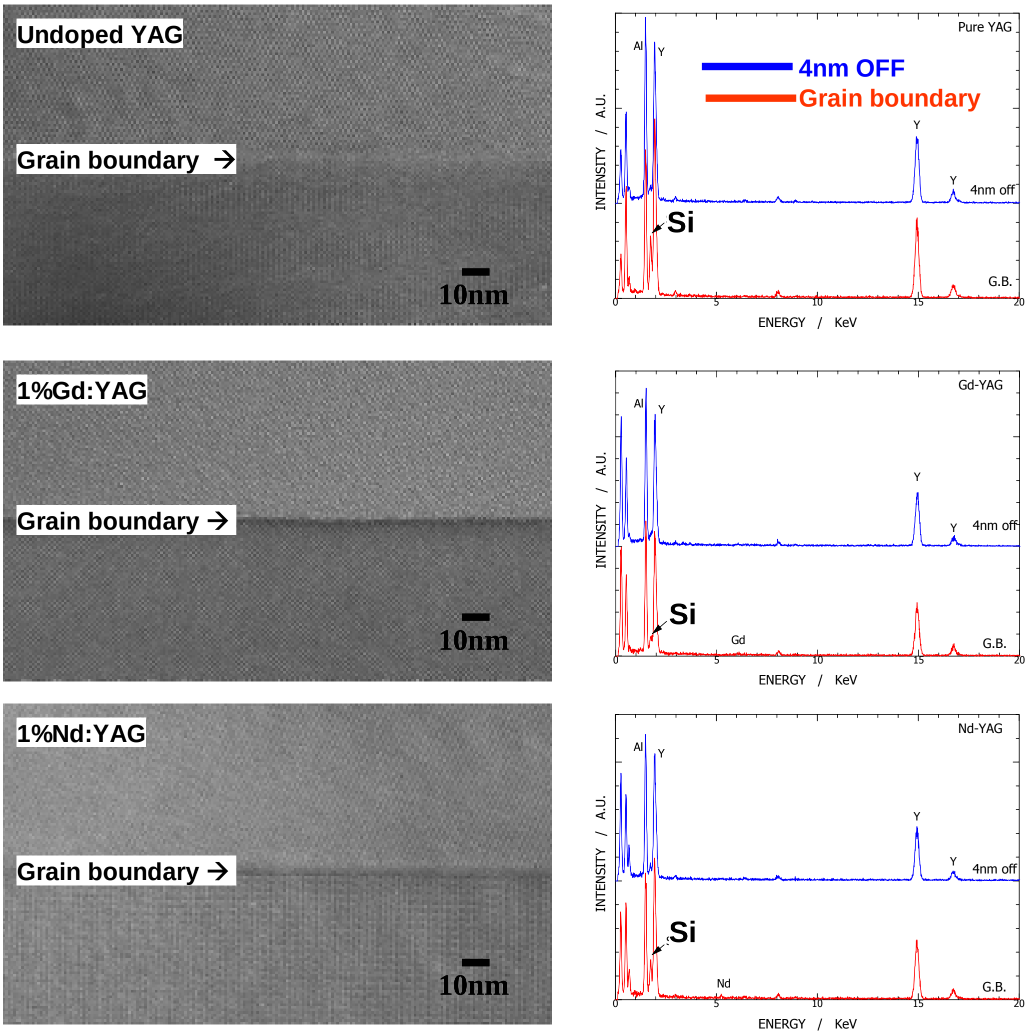


Fig.12 (Left) High resolution transmission electron microscopy (HR-TEM) images and (right) energy-dispersive spectroscopy (EDS) analysis results of the fabricated ceramic samples.

8. Conclusions

High optical quality transparent undoped YAG, 1%Gd:YAG, and 1%Nd:YAG ceramic element were successfully fabricated. Uniform microstructures were confirmed and the average grain size was approximately 10μm. The in-line transmittance of each sample was comparable to that of the commercial single crystal Nd:YAG, and the transmittance at 1064nm was 84%, a theoretical value. Refractive index distribution was confirmed to be very homogeneous by interferometry image, showing straight fringes. Almost no residual pores were recognized under optical microscope and SEM observation. The quality of the optical ceramic gain media was found to improve under macro and microscopic observation. In addition, HR-TEM and EDS analysis results revealed that the fabricated ceramic samples have almost no atomic defects.

9. Future plan

Fabrication process will be optimized to achieve ceramic laser gain media with much higher quality especially on milling condition, granulation condition, CIP and HIP condition. Optical characterizations by applying a light scattering tomography by He-Ne laser, and investigation of laser induced damage by irradiating with a high energy pulsed laser will be performed. In addition, simple laser oscillation test will be performed and their laser performance will be compared with the Konoshima ceramic and Nd:YAG single crystal.

Meanwhile, fabrication of waveguide laser elements with simple waveguide layer and with composite waveguide layers (three-layered and five-layered core, refer Fig.13) will be tested by new ceramic bonding technology. Then, the developed laser gain media will be tested for the possibility of high power laser oscillation test from 100W to 1kW output level.

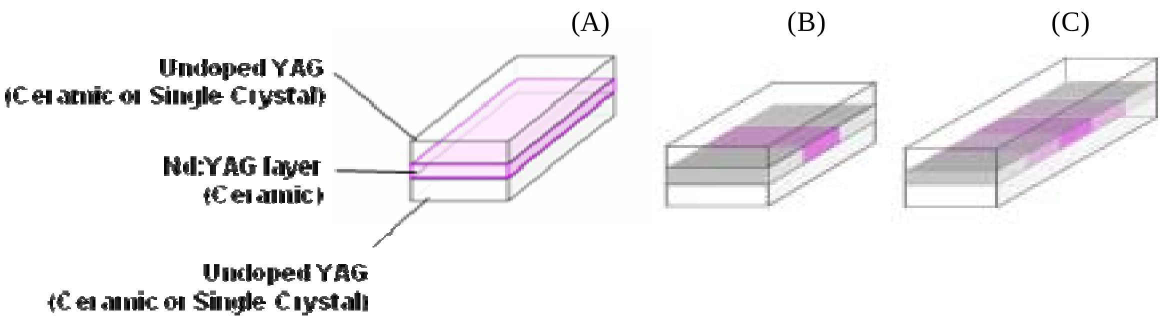


Fig.13 (A) Simple waveguide element, (B) Advanced waveguide with three-layered core, and (C) Advanced waveguide with five-layered core.

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